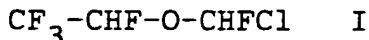


Translation of German Application # 2,361,058

1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether and Method for its Production

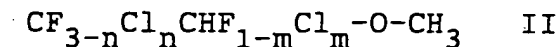
The object of the invention is the

1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether of the formula:



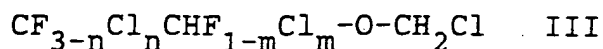
as well as methods for its production, which are characterised in that:

A: a 1.2.2.2-tetrahalogenethyl-methyl-ether of the general formula



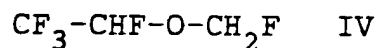
where n can take the values 0 to 3 and m = 0 or 1, is subjected to a partial photochlorination to form the corresponding

1.2.2.2-tetrahalogenethyl-chloro-methyl-ether of the general formula



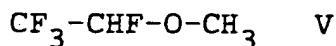
in which n and m have the same meaning as in formula II, the obtained compound of formula III is fluorinated in

1.2.2.2-tetrafluoroethyl-fluoromethyl-ether of the formula

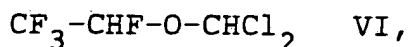


the compound of formula IV is again chlorinated under light exposure partially to 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether of formula I, and the obtained ether is isolated from the chlorination mixture by usual methods.

B. the 1.2.2.2-tetrahalogenethyl-methyl-ether of formula II, wherein  $n = 0$  and  $m = 0$ ,



is reacted by partial photochlorination into 1.2.2.2-tetrafluoroethyl-dichloromethyl-ether of the formula:

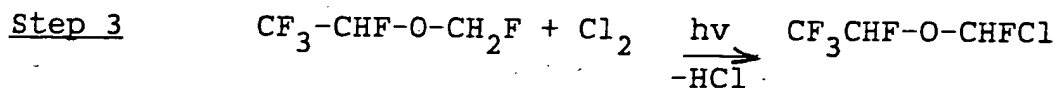
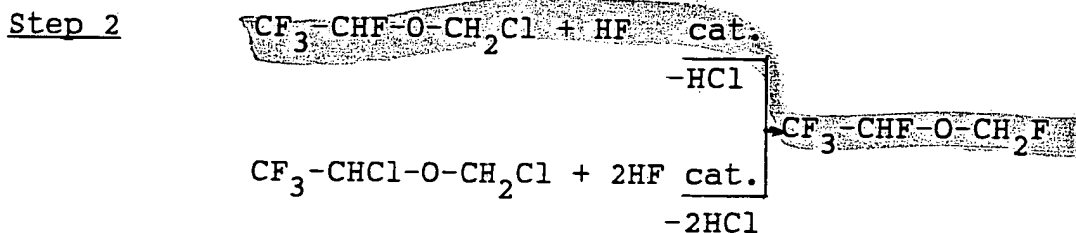
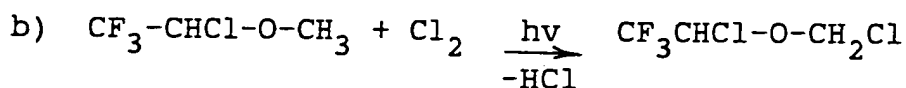
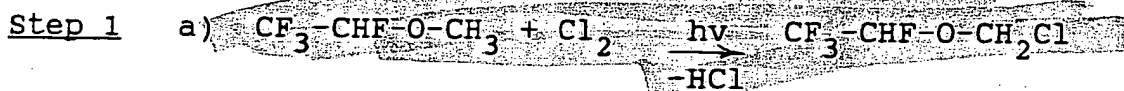


the latter is then partially fluorinated to form the 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether (I) and this ether is isolated from the fluorination mixture in a usual manner.

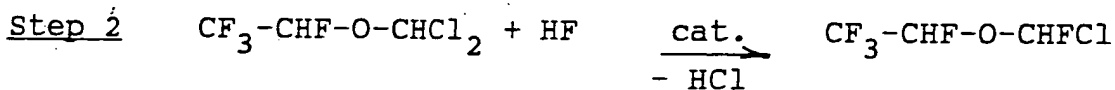
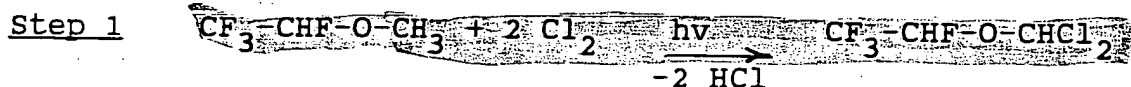
Subjects of the invention are also inhalation-anesthetics which are characterised by comprising 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether, as well as the use of the 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether as inhalation-anesthetic.

The methods for the production of the 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether (I) comprise the stepwise selective chlorination and fluorination of the starting compounds  $\text{CF}_3\text{-Cl}_n\text{-CHF-Cl}_m\text{-O-CH}_3$  (II). They can be reproduced for example for the preferred case  $n = 0$  and  $m = 0$  or  $1$  by the following equation sequences:

Operation mode A:



Operating mode B:



The starting materials for both operating modes for the

production of  $\text{CF}_3\text{-CHF-O-CHFCl}$  (I), which have the general formula  $\text{CF}_3\text{-}_n\text{Cl}_n\text{-CHF}_1\text{-}_m\text{Cl}_m\text{-O-CH}_3$  (II) where  $n$  and  $m$  have the meaning mentioned before, can be produced by substitution of the free OH-group of methylhemiacetals of perhalogenacetaldehyde  $\text{CCl}_3\text{CHO}$ ,  $\text{CCl}_2\text{F-CHO}$ ,  $\text{CClF}_2\text{-CHO}$  and preferably  $\text{CF}_3\text{-CHO}$  for the case  $m = 0$ , with fluorine, especially with the help of (2-chloro-1.1.2-trifluoro-ethyl)-dialkylamines according to German patent application P 23 40 560.9, for the case  $m = 1$  with chlorine by means of acid chlorides, preferably phosphorpentachloride according to German patent application P 23 40 561.0.

The reaction with chlorine in the method steps A/1, A/3 and B/1 should preferably take place in the  $\text{C}_1$ -group of the ethyl-methyl-ether II, IV and V. The provision for achieving the desired selectivity is the selection of photochlorination as operation mode under comparatively mild thermal conditions. As light source one can use all those which are known for this purpose, i.e. such sources which emit sufficient short wave light for activation of the chlorine, such as for example incandescent lamps, UV-lamps, mercury lamps or even strong sun light. The form of the illumination depends on the material of the used chlorination vessel. In case of use of opaque vessels resistant to chlorine and hydrogen chloride, for example of nickel, nickel alloys, steel, steel alloys, porcellane or ceramic, the illumination will be selected for example by means of a dipping lamp, in case of use of transparent vessel material the illumination from the exterior will be generally selected.

As further provisions for increasing the selectivity of the photochlorination described in the above equations use can be made of the removal of the reaction heat by external

cooling, of the fine distribution of the chlorine flow by the use of an introduction device comprising a frit, by the stirring of the liquid phase of the ethyl-methyl-ether to be chlorinated having the formula II, IV or V and/or by the addition of a solvent means inert against chlorine and hydrogen chloride, such as for example  $\text{CCl}_4$ , as well as by the dilution of the chlorine flow with inert gasses, for example with hydrogen chloride. They can be used separately or in all possible combinations.

The preferred embodiment for the reaction steps A/1, A/3 and B/1 consists of introducing undiluted gaseous chlorine through a frit into the liquid phase of the ethyl-methyl-ether of formula II, IV or V, which is contained without solvent or diluent in a glass vessel with outer cooling at a selected temperature and is illuminated from the exterior by means of a strong light source.

The reaction temperature and the necessary chlorine quantity can be different in the reaction steps A/1, A/3 and B/1.

For the production of 1.2.2.2-tetrahalogenethyl-chloromethyl-ether of formula III with  $m = 0$  (operation mode A, step 1a) by photochlorination of the starting compounds having the formula II with  $m = 0$ , the reaction temperature can be in the range between  $-10^\circ$  and  $+50^\circ\text{C}$ . For general technical reasons, this temperature will be especially selected between  $0^\circ$  and  $+40^\circ\text{C}$ , preferably between  $+5^\circ$  and  $+20^\circ\text{C}$ .

The quantity of the introduced chlorine is maintained, in order to avoid overchlorination, below the stoichiometrically necessary equimolar quantity, that means preferably below 0.9 mol, especially between 0.5 and 0.8 mol chlorine per mol of ether of formula II. It is possible to

go below the lower limit of 0.5 mol, it is simply preferred for practical considerations as regards the distillation costs and the distillation losses.

The corresponding chloromethyl-ether  $\text{CF}_{3-n}\text{Cl}_n\text{-CHF-O-CH}_2\text{Cl}$  with  $n = 0$  to 3, can be separated from the chlorination product, optionally after the usual wash and drying steps, by fractional distillation, with a good yield and in a pure form.

The production of the intermediate products of the general formula III with  $m = 1$  (operation mode A, step 1b), of the ether  $\text{CF}_{3-n}\text{Cl}_n\text{-CHCl-O-CH}_2\text{Cl}$  with  $n = 0$  to 3 can be effected as described above for the case  $m = 0$  or in accordance with patent application P 23 44 442.0.

In step 2 of the operation mode A the chloromethyl-ethers of the general formula III are fluorinated preferably with hydrogen fluoride in the presence of a fluorination catalyst, especially according to one of the known processes of gas phase fluorination over a fixed bed catalyst, for example aluminium fluoride or preferably chromoxyfluoride, according to the so-called antimony method (antimony(V)chlorofluoride + HF), wherein antimony can be replaced by arsenic in a known manner, or by reaction with a known fluorination agent such as for example  $\text{SbF}_5$ .

The reaction temperature for the preferred catalysed gas phase process with the use of hydrogen fluoride has a lower limit at the temperature at which the catalyst starts to be active, and has an upper limit at the decomposition temperature of the ether of formula III to be fluorinated and of the 1.2.2.2-tetrafluoroethyl-fluoromethyl-ether (IV) on the surface of the catalyst. In a suitable manner a value between  $80^\circ$  and  $220^\circ \text{C}$ , preferably between  $100^\circ$  and  $170^\circ \text{C}$ ,

especially between 120° and 150 °C will be maintained as fluorination temperature.

The quantity of hydrogen fluoride per mol  $\text{CF}_3\text{-Cl}_n\text{-CHF}_1\text{-Cl}_m\text{-O-CH}_2\text{Cl}$  (III) in the gas phase method is at least  $(1+m+n)$  mol. For the completion of the reaction, for its acceleration and for a slight vaporisation of the ether of formula III one will, however, generally operate with an excess of HF up to 10  $(1+m+n)$  mol, preferably between 3  $(1+m+n)$  and 7  $(1+m+n)$  mol.

The 1.2.2.2-tetrafluoroethyl-fluoromethyl-ether (IV) can be isolated from the fluorination product, optionally after aqueous treatment for elimination of HF and HCl and drying, by fractional distillation in pure form.

The 3. reaction step of the operation mode A yields finally the claimed ether  $\text{CF}_3\text{-CHF-O-CHFCl}$  by photochlorination of  $\text{CF}_3\text{-CHF-O-CH}_2\text{F}$  according to the method and apparatus conditions mentioned above. The chlorination is effected at a temperature between 0 °C and the boiling point of  $\text{CF}_3\text{-CHF-O-CH}_2\text{F}$ , preferably between 20 °C and 40 °C.

In order to permit the partial chlorination, i.e. the introduction of 1 gram-atom chlorine per mol  $\text{CF}_3\text{-CHF-O-CH}_2\text{F}$ , and to largely prevent overchlorination, chlorine will be generally introduced in a quantity lower as equimolar, preferably at most 0.9 mol, for practical reasons, however, at least 0.4 mol, especially a quantity between 0.5 and 0.8 mol per mol of 1.2.2.2-tetrafluoroethyl-fluoromethyl-ether (IV) in the reaction.

The isolation of the ether according to the invention having the formula  $\text{CF}_3\text{-CHF-O-HFCl}$  from the chlorination product of

the reaction step A/3 can be effected in a usual manner after washing and drying steps by fractional distillation in a good yield. According to the effectiveness of the fractionation, the  $\text{CF}_3\text{-CHF-O-CHFCl}$  can contain as impurities small quantities of compounds having similar boiling points. Besides the fractional distillation, the separation of  $\text{CF}_3\text{-CHF-O-CHFCl}$  from the chlorination product can be effected with a very good success by preparative gas chromatography.

The 1.2.2.2-tetrafluoroethyl-methylether of formula V serves as starting product for the mode of operation B of the synthesis of the ether of formula I according to the invention, in which two chlorine atoms are selectively introduced into the methyl group by means of photochlorination. The step B/1 is again carried out under method and apparatus conditions mentioned in connection with the operating mode A/1. The chlorination is generally carried at a temperature between  $-10^\circ$  and  $+50^\circ\text{C}$ , preferably between  $0^\circ$  and  $+40^\circ\text{C}$ , especially between  $+5^\circ$  and  $+20^\circ\text{C}$ . It can be continued as long as 2 mol chlorine are used for each mol of  $\text{CF}_3\text{-CHF-O-CH}_3$ . In order to avoid the formation of higher quantities of higher chlorinated by-products, it is advisable to react less than the double molar quantity, preferably 1.65 to 1.8 mol per mol of 1.2.2.2-tetrafluoroethylmethylether (V).

The chlorination of the compound of formula V into the ether of formula I can also be effected in two steps, such that one chlorine atom is firstly introduced into the methyl group of (V) according to step 1a of the operation mode A, the obtained 1.2.2.2-tetrafluoroethyl-chloromethyl-ether is isolated by fractional distillation and only then is the second chlorine atom introduced into the chloromethyl group to form (VI). In each chlorination step it is here preferred



to use 0.5 to 0.8 mol chlorine per mol ether.

The intermediate product  $\text{CF}_3\text{-CHF-O-CHCl}_2$  can be purified by fractional distillation, suitably after previous washing and drying steps.

The partial fluorination of  $\text{CF}_3\text{-CHF-O-CHCl}_2$  into the claimed ether  $\text{CF}_3\text{-CHF-O-CHFCl}$  (step 2 of the operation mode B) can be effected as well with hydrogen fluoride in the presence of a fluorination catalyst, such as for example  $\text{SbF}_{5-n}\text{Cl}_n$  ( $n = 0$  to 5), arsenic (V)-chloro-fluoride or  $\text{SnCl}_4$ , as well as with  $\text{SbF}_3$  in the presence of  $\text{SbCl}_5$ . Hydrogen fluoride is introduced in gaseous form or poured dropwise in a liquid form, or  $\text{SbF}_3$  is introduced in solid form into the mixture of ether (VI) with the catalyst, which is contained in a reaction vessel of nickel, steel or copper. The introduction of gaseous HF is preferred.

This liquid phase fluorination is carried out at a temperature of  $-70^\circ$  to  $+20^\circ \text{C}$ , preferably between  $-50^\circ$  and  $0^\circ \text{C}$ , especially  $-30^\circ$  to  $-10^\circ \text{C}$ . At higher temperatures the reaction remains not sufficiently selective on the monofluorination step.

In addition to the temperature the quantity of metered hydrogen fluoride also contributes to attain the step of the chlorofluoromethylether. Suitably, less than the equimolar quantity of HF per mol  $\text{CF}_3\text{-CHF-O-CHCl}_2$  will be introduced, preferably a quantity between 0.4 and 0.8 mol HF per mol ether (VI).

The ether of formula I according to the invention can be attained in a pure form in a usual manner, for example by washing the fluorination product with water in order to eliminate acids, by drying and by final fractional

distillation.

In all reaction steps of both alternative operation modes A and B for the production of  $\text{CF}_3\text{-CHF-O-CHFCl}$  (I) in which the reaction is not continued until the stoichiometric conversion in order to avoid by-products which are not useful for the synthesis of (I), the yield of useful intermediate products, or  $\text{CF}_3\text{-CHF-O-CHFCl}$ , can be increased by subjecting the non-reacted starting compounds of the respective reaction step, which can easily be separated by fractional distillation, again to this reaction.

The 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether,  $\text{CF}_3\text{-CHF-O-CHFCl}$ , under normal conditions is a readily flowing, colorless liquid as clear as waterwhite liquid having a slight but agreeable smell. It is characterised by the following physical properties: Boiling point at 760 mm:  $51.5^\circ\text{C}$ , refraction index  $n_D^{25} = 1.2998$ , density at  $25^\circ\text{C}$ : 1.5234, molecular weight: 184.5. It is a very effective inhalation anesthetic, non-flammable and stable against so-called breathing lime which comprises for example a mixture of  $\text{Ba(OH)}_2$  and  $\text{Ca(OH)}_2$ . The compound is further slightly mixable with other organic liquids, and shows advantageous properties as solvent for fluorinated olefines, other fluorinated substances, greases and oils and can be used on account of this property as cleaning agent for example for metal surfaces.

The 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether according to the invention acts as a narcotic when added to the breathing air of living organisms and is usable for this reason as inhalation anesthetic. Owing to its relatively low boiling point it can be easily and in a controlled manner admixed to breathing mixtures which permit the maintenance of life during the narcose by sufficient oxygen

concentrations.

The ether according to the invention can also be used together with other inhalation anesthetics such as for example laughing gas or diethylether, or together with other anesthetic and therapeutical auxiliaries like for example muscle relaxants, barbiturates and plasma extenders, as it is usually necessary in modern combination narcose.

The effect of the

1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether as inhalation narcotic will become clear from the results of a pharmacological test which has been carried out as follows: Groups of four mice have been placed in a glas bell containing 26 liters of a narcose mixtures, which were produced by vaporisation of  $\text{CF}_3\text{-CHF-O-CHFCl}$  in different quantities. The animals were kept each time for a duration of 10 minutes in the  $\text{CF}_3\text{-CHF-O-CHFCl}$ /air atmosphere. The course of the narcose as well as the wake up of the animals have been observed.

$\text{CF}_3\text{-CHF-O-CHFCl}$  brings only a slight excitation during the beginning of the narcose, leads already with small concentration to a status of tolerance and is characterised by very short wake-up time. Its narcose width - the difference between the lethal concentration and the concentration which is necessary for maintaining a tolerance status, expressed by the ratio of both concentrations - amounts to 4 and it is therefore an inhalation anesthetic of good use.

The following Table 1 shows the times  $t_I$  until appearance of the tolerance stage and the times  $t_{II}$  until waking up of the mice after suppression of the narcose means as a function of the concentration of the narcose means (ml of vaporised

liquid per 26 l or air) from a comparable series of tests, in which the ether  $\text{CF}_3\text{-CHF-O-CHFCl}$  according to the invention is compared to the isomers available on the market 1-chloro-2.2.2-trifluoroethyl-difluoromethylether ( $\text{CF}_3\text{-CHCl-O-CHF}_2$ , A) and 2-chloro-1.1.2-trifluoroethyl-difluoromethylether ( $\text{CHFCl-CF}_2\text{-O-CHF}_2$ , B). (Table 1 is to be found at page 13).

It can be clearly seen from the values of table (1) that the ether according to the invention of formula  $\text{CF}_3\text{-CHF-O-CHFCl}$  possesses advantageous properties over the two isomers A and B as regards the use as inhalation anesthetics. It is remarkable on the one hand that the tolerance status can be attained with it at lower active concentrations as with the isomer B, on the other hand it is apparent that the recovery time  $t_{\text{II}}$  is significantly shortened in comparison to that of isomer A in the interesting range of lower active concentrations. The fast reaching of the tolerance status at lower concentration of narcose agent and the speed of decay of the narcose after its interruption are advantageous properties for an inhalation anesthetics which provide a reduction in the risk of damaging side effects on the heart muscles and the parenchymatic organs, especially the liver, and the avoidance of acute narcose accidents. The combination of this both properties in the 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether according to the invention represents an improvement towards the ideal narcose agent.

The method for production of the 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether will be explained by means of the following examples of production. In the examples the intermediate samples of the destillation have not been taken into account.

Table 1

| Substance   | 1,5 ml/26 l    |                 | 1,75 ml/26 l   |                 | 2,0 ml/26 l    |                 | 2,5 ml/26 l    |                 | 5,0 ml/26 l    |                 |
|---|----------------|-----------------|----------------|-----------------|----------------|-----------------|----------------|-----------------|----------------|-----------------|
|   | t <sub>I</sub> | t <sub>II</sub> | t <sub>I</sub> | t <sub>II</sub> | t <sub>I</sub> | t <sub>II</sub> | t <sub>I</sub> | t <sub>II</sub> | t <sub>I</sub> | t <sub>II</sub> |
| CF <sub>3</sub> -CHF-O-CHFCl                                      | 5'             | <15"            | 2'50"          | 40"             | 2'50"          | 50"             | 1'45"          | 2'20"           | 2'             | 3'20"           |
| A CF <sub>3</sub> -CHCl-O-CHF <sub>2</sub><br>(comparative agent) | 1'50"          | 1'50"           |                |                 | 1'50"          | 2'5"            |                |                 | 1'             | 5'30"           |
| B CHFCl-CF <sub>2</sub> -OCHF <sub>2</sub><br>(comparative agent) |                |                 |                |                 | 6'30"          | 50"             | 1'50"          | 2'50"           | 1'45"          | 5'20"           |

### EXAMPLES OF PREPARATION

#### Example 1: Preparation of $\text{CF}_3\text{-CHF-O-CH}_2\text{Cl}$

In a cylindrical chlorination vessel having an inlet tube and a frit connected thereto as well as two tubes for a low temperature cooler and a thermometer are placed 1945 g (14,8 mols) of 1.2.2.2-tetrafluoroethyl-methyl-ether and cooled to a temperature of 10 °C. Under illumination from the exterior by means of a 200 Watt lamp and at 9° to 16 °C 875 g (12,3 mols) of chlorine are introduced as rapidly as it is consumed. The resulting hydrogen chloride escapes through the low temperature cooler (- 70 °C) and is absorbed in water (11,21 mols). After termination of the chlorination the reaction product is successively washed with a sodium hydrogen sulfite solution, with water and with sodium hydrogen carbonate solution and dried over  $\text{MgSO}_4$ . The dried crude product (2129 g) is fractionally distilled, with the result that besides to 662 g of pure starting material  $\text{CF}_3\text{CHF-O-CH}_3$  a fraction of 1097 g  $\text{CF}_3\text{-CHF-O-CH}_2\text{Cl}$  with a purity over 99,3% is obtained. Boiling point 63°C/754 mm.

$\text{CF}_3\text{-CHF-O-CH}_2\text{Cl}$       MG 166,5

calculated values: C 21,6%; H 1,8%; F 45,6%; Cl 21,3%

actual values:      C 21,7%; H 1,8%; F 45,4%; Cl 21,3%

#### Example 2: Preparation of $\text{CF}_3\text{-CHF-O-CH}_2\text{F}$ from $\text{CF}_3\text{-CHF-O-CH}_2\text{Cl}$

Through a vertically oriented nickel tube reactor, which is electrically heated from the exterior and contains a chromoxifluoride catalyst (produced according to German patent 1 252 182) with a bulk volume of 900 ml, 2872 g (17,26 mols) of 1.2.2.2-tetrafluoroethyl-chloromethyl-ether, after passing through a vaporisator, together with 2150 g

(107,5 mols) of hydrogen fluoride are passed during 17 hours at an inner temperature of 130° - 140 °C. After leaving the reaction region, the reaction gasses are absorbed in ice water such that the organic product separates as an own phase, whereas HF and HCl dissolve in water. The organic crude product (2075 g) washed and dried over  $\text{MgSO}_4$  is fractionally distilled. A fraction of 1501 g of pure 1.2.2.2-tetrafluoroethyl-fluoromethyl-ether having a boiling point of 42 °C - 42,5 °C/750 mm and  $n_D^{20} < 1.3000$  is obtained, in addition to 289 g of non-reacted  $\text{CF}_3\text{-CHF-O-CH}_2\text{Cl}$ .

$\text{CF}_3\text{-CHF-O-CH}_2\text{F}$             MG 150

Calculated values: C 24,0%; H 2,0%; F 63,3%

actual values:            C 23,8%; H 2,1%; F 62,8%

Example 3: Preparation of  $\text{CF}_3\text{-CHF-O-CH}_2\text{F}$  from  
 $\text{CF}_3\text{-CHCl-O-CH}_2\text{Cl}$

Through an electrically heated nickel tube reactor, filled with a chromoxifluoride catalyst as in example 2, a gaseous mixture of 343 g (1,875 mols) of 1-chloro-2.2.2-trifluoroethyl-chloromethyl-ether (produced according to German patent application P 23 44 442.0) and 470 g (23,5 mols) hydrogen fluoride are introduced during 3 hours at a temperature of 125 °C to 135 °C. The reaction gasses leaving the reactor are absorbed in ice water, such that the organic product separates. After washing with water and drying with  $\text{MgSO}_4$ , the product (192 g) is fractionally distilled. Besides 12 g starting compound  $\text{CF}_3\text{-CHCl-O-CH}_2\text{Cl}$  106 g  $\text{CF}_3\text{-CHF-O-CH}_2\text{F}$

(boiling point 44 °C/765 mm) and 68 g of  $\text{CF}_3\text{-CHF-O-CH}_2\text{Cl}$  (boiling point 63 °C - 64 °C/764 mm) are also isolated.

Example 4: Preparation of  $\text{CF}_3\text{-CHF-O-HFCl}$  from  $\text{CF}_3\text{-CHF-O-CH}_2\text{F}$

In a chlorination vessel having an inlet tube, a low temperature cooler and a thermometer 705 g (4,7 mols) of 1.2.2.2-tetrafluoroethyl-fluoromethyl-ether are placed and heated to the boiling stage. Through the frit of the inlet tube 220 g (3,1 mol) of chlorine are introduced at 40°-41 °C and under illumination with a 200 Watt lamp, as rapidly as it is consumed. The resulting hydrogen chloride after escaping through the cooler is absorbed in water and determined by titration (2,84 mols). After termination of the chlorination, the product is treated with sodium hydrogen sulfit solution, with water and with sodium hydrogen carbonate solution and finally dried over  $\text{MgSO}_4$ . With the help of preparative gas chromatography (stationary phase: GE-XE 60, 25% cyanoethyl-, 75% methylpolysiloxane), besides 332 g of non-transformed  $\text{CF}_3\text{-CHF-O-CH}_2\text{F}$ , 232 g of pure  $\text{CF}_3\text{-CHF-O-CHFCl}$  having a boiling point of 51,5 °C/760 mm are isolated.

$\text{CF}_3\text{-CHF-O-CHFCl}$  MG 184.5

Calculated values: C 19,5%; H 1,1%; F 51,4%; Cl 19,2%

Actual values: C 19,8%; H 1,2%; F 51,0%; Cl 19,2%

Example 5: Preparation of  $\text{CF}_3\text{-CHF-O-CHCl}_2$

In a chlorination vessel made of glass, which comprises an introduction tube having a frit, a low temperature cooler and a thermometer, 950 g (7,2 mol) of  $\text{CF}_3\text{-CHF-O-CH}_3$  are cooled to a temperature of 10 °C. Under illumination with a 200 Watt lamp 1085 g (15,3 mol) of chlorine are introduced



during 21 hours at this temperature. The produced hydrogen chloride leaves the reaction vessel through the cooler and is absorbed in water (15,28 mol). After treatment of the chlorination product by washing with sodium hydrogen sulfite solution, water and sodium hydrogen carbonate solution as well as drying over  $\text{MgSO}_4$ , this product is fractionally distilled. From 1203 g of dry crude product the following pure fractions, in addition to intermediate fractions are obtained:

1) 137 g, boiling point:  $63 - 66^\circ\text{C}/763\text{ mm}$ ,  $\text{CF}_3\text{-CHF-O-CH}_2\text{Cl}$   
(purity 95%)

2) 583 g, boiling point:  $81 - 82^\circ\text{C}/750\text{ mm}$ ,  $\text{CF}_3\text{-CHF-O-CHCl}_2$   
(purity 95%)

$\text{CF}_3\text{-CHF-O-CHCl}_2$  <sup>l<sub>2</sub></sup> MG 201

Calculated values: C 17,9%; H 1,0%; F 37,8%; Cl 35,4%

Actual values: C 17,7%; H 1,0%; F 37,3%; Cl 36,1%

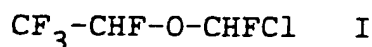
Example 6: Preparation of  $\text{CF}_3\text{-CHF-O-CHFCl}$  from  
 $\text{CF}_3\text{-CHF-O-CHCl}_2$

In a 1 liter copper vessel having a lid, which comprises openings for a thermometer, a reflux condenser and an introduction tube, the latter being also made of copper, are placed 508 g (2,5 mol) of 1.2.2.2-tetrafluoroethyl-dichloromethyl-ether and 10 g  $\text{SbCl}_5$  as catalyst and cooled to a temperature of  $-25^\circ\text{C}$ . Within two hours and at this temperature 44 g (2,2 mol) of hydrogen fluoride in gaseous form are introduced. The hydrogen fluoride which escapes through the copper cooler maintained at  $0^\circ\text{C}$  is absorbed in water and determined by titration (2,13 mol). After termination of the introduction of HF, the fluorination product is heated to  $0^\circ\text{C}$  and placed

on ice, the organic phase is separated, washed with water and dried over  $\text{MgSO}_4$ . From the crude product (348 g) besides 88 g of  $\text{CF}_3\text{-CHF-O-CHF}_2$  (boiling point  $24,5^\circ\text{C}/764\text{ mm}$ ) 124 g of non-reacted  $\text{CF}_3\text{-CHF-O-CHCl}_2$  (boiling point  $80,5^\circ\text{-}82^\circ\text{C}/760\text{ mm}$ ), 95 g of  $\text{CF}_3\text{-CHF-O-CHFCl}$  having a boiling point of  $51^\circ\text{-}53,5^\circ\text{C}/764\text{ mm}$  are isolated by fractional distillation.

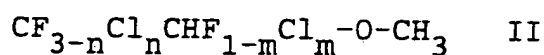
Patent Claims:

1. 1.2.2.2-Tetrafluoroethyl-chlorofluoromethyl-ether of the formula

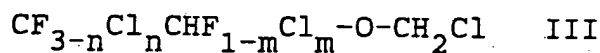


2. Method for the production of the 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether characterised in that:

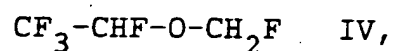
- A. a 1.2.2.2-tetrahalogenethyl-methyl-ether of the general formula



wherein n can be 0 to 3 and m is 0 or 1, is subjected to a partial photochlorination to form of the corresponding 1.2.2.2-tetrahalogenethyl-chloromethyl-ether of the general formula



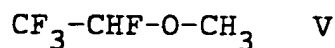
wherein n and m have the meaning mentioned in connection with formula II, the obtained compound of formula III is fluorinated to 1.2.2.2-tetrafluoroethyl-fluoromethyl-ether of the formula



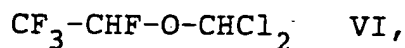
the compound of formula IV is again partially chlorinated under illumination to

1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether of formula I and the obtained ether is isolated according to usual methods from the chlorination mixture, or

- B. the 1.2.2.2-tetrahalogenethyl-methylether of formula II, where  $n = 0$  and  $m = 0$ ,

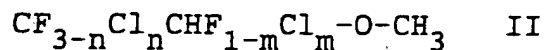


is reacted by partial photochlorination into 1.2.2.2-tetrafluoroethyl-dichloromethyl-ether of formula



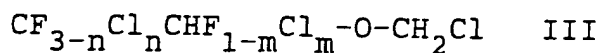
the latter is then partially fluorinated to form of the 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether (I) and this ether is isolated in usual manner from the fluorination mixture.

3. Method according to claim 2, characterised in that a 1.2.2.2-tetrahalogenethyl-methyl-ether of the general formula



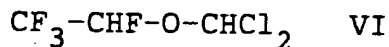
where  $n = 0$  and  $m = 0$  or 1 is partially photochlorinated.

4. Method according to claim 2, characterised in that the 1.2.2.2-tetrahalogenethyl-chloromethyl-ether of the general formula



is fluorinated with hydrogen fluoride in the presence of a fluorination catalyst.

5. Method according to claim 2, characterised in that the 1.2.2.2-tetrafluoroethyl-dichloromethyl-ether of formula



is fluorinated with hydrogen fluoride in the presence of a fluorination catalyst.

6. Inhalation anesthetics, characterised by comprising 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether as agent.
7. Inhalation anesthetics according to claim 6, characterised by comprising a mixture of 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether with one or several usual inhalation narcotics.
8. Inhalation anesthetics according to claim 7, characterised by comprising a mixture of 1.2.2.2-tetrafluoroethyl-chlorofluoromethyl-ether and laughing gas.
9. Inhalation anesthetics according to claims 6 to 8, characterised by comprising an amount of oxygen sufficient for maintaining life.
10. Use of the 1.2.2.2-tetrafluoroethyl-chlorofluoroethyl-ether as inhalation anesthetic on living organisms capable of

being narcotised.